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**IS : 8050 - 1976**

*Indian Standard*  
**SPECIFICATION FOR ALPHA PICOLINE**

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**INDIAN STANDARDS INSTITUTION**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

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**AMENDMENT NO. 1    FEBRUARY 1989**  
**TO**  
**IS : 8050 - 1976   SPECIFICATION FOR**  
**ALPHA PICOLINE**

[ *Page 4, Table 1, col 3, Sl No. (i)* ] — Substitute ' 98·0 ' for ' 98 '.

[ *Page 4, Table 1, col 3, Sl No. (ii)* ] — Substitute ' 128·0 to 131·0°C ' for ' 128 to 131 °C '.

[ *Page 4, Table 1, col 4, Sl No. (ii)* ] — Substitute ' 127·0 to 132·0°C ' for ' 127 to 132°C '.

( PCDC 9 )

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# Indian Standard

## SPECIFICATION FOR ALPHA PICOLINE

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# *Indian Standard*

## SPECIFICATION FOR ALPHA PICOLINE

### 0. FOREWORD

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 6 May 1976, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

**0.2** Alpha picoline is used in the manufacture of picolinic acid, 2-vinyl pyridine, 2-pyridylethanol, 2 beta-methoxyethyl pyridine and pyridine 2-aldehyde. It also finds use in the dyestuff industry as a solvent.

**0.3** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS:2-1960\*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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### 1. SCOPE

**1.1** This standard prescribes the requirements and the methods of sampling and test for alpha picoline.

### 2. GRADES

**2.1** There shall be two grades of the material, namely

- a) *Grade 1* — suitable for use in pharmaceutical industry and other industries, and
- b) *Grade 2* — for use as solvent in dyestuff industry.

### 3. REQUIREMENTS

**3.1 Description** — The material shall be a clear liquid having a strong characteristic odour.

**3.2 Solubility** — The material shall pass the test as prescribed in A-5.

**3.3** The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of the appendix is given in col 5 of the table.

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\*Rules for rounding off numerical values ( revised ).



TABLE 1 REQUIREMENTS FOR ALPHA PICOLINE

( Clause 3.3 )

Sl No	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST ( REF TO CL No IN APPENDIX A)
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Alpha picoline content, percent by mass, <i>Min</i>	98	96.5	A-2
ii)	Boiling range at 760 mm-Hg:	2 to 97 percent by volume shall distil between a range of 128 to 131°C	2 to 97 percent by volume shall distil between a range of 127 to 132°C	A-3
iii)	Moisture content, percent by mass, <i>Max</i>	0.2	0.4	A-4

#### 4. PACKING AND MARKING

**4.1** The material shall be packed in mild steel drums. The gaskets for the bungs shall be of high density polyethylene.

**4.2 Marking** — The containers shall be suitably marked with the following information:

- a) Name and grade of the material.
- b) Net mass of the material in the container.
- c) Name of the manufacturer and his recognized trade-mark, if any; and
- d) Batch number or lot number, in code or otherwise.

##### 4.2.1 The containers may also be marked with the ISI Certification Mark

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

## 5. SAMPLING

**5.1** The procedure for sampling and the criteria for conformity of the material shall be as prescribed in Appendix B.

# APPENDIX A

( Clause 3.2, and Table 1 )

## METHODS OF TEST FOR ALPHA PICOLINE

### A-1. QUALITY OF REAGENTS

**A-1.1** Unless specified otherwise, pure chemicals and distilled water ( *see* IS : 1070-1960\* ) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

### A-2. DETERMINATION OF ALPHA PICOLINE

**A-2.0 Outline of the Method** — The content of alpha picoline alongwith other components is determined by gas chromatography.

#### A-2.1 Apparatus

**A-2.1.1 Gas Chromatograph** — with flame-ionization detector.

**A-2.1.2 Potentiometric Strip Chart Recorder** — full scale deflection, 1 second ( preferably provided with either a mechanical disc or electronic integrator ).

**A-2.1.3 Temperature Programmer**

**A-2.1.4 Column** — of stainless steel or glass, 1.8 m long and 3.0 mm in diameter, packed with 15 to 20 percent of carbowax 400 or 1500 on chromosorb W 150-180 micron.

#### A-2.2 Tests Substances

**A-2.2.1 Alpha, Beta and Gamma Picolines** ( *Pure Grades* )

**A-2.2.2 Standard for Analysis** — xylene 1.0 percent, pyridine 1.0 percent, alpha picoline 97.0 percent and gamma picoline 1.0 percent.

#### A-2.3 Procedure

**A-2.3.1 Operating Parameters of Gas Chromatograph**

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\*Specification for water, distilled quality ( *revised* ).

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**A-2.3.1.1 Injection temperature** — 150°C to 180°C.

**A-2.3.1.2 Column temperature** — 80°C to 100°C.

**A-2.3.1.3 Carrier gas** — Nitrogen (99.99 percent purity v/v). Flow rate 30 ml/min. Combustible gases: Hydrogen, 30 ml/min and compressed air: 300 ml/min.

**A-2.3.1.4 Chart speed**

<i>Slow</i>	<i>Medium</i>	<i>Fast</i>
75 cm/h	150 cm/h	300 cm/h

**A-2.3.1.5 Standard sample** — Introduce about 0.5 microlitre of standard sample into the chromatograph.

**A-2.3.1.6 Detector temperature** — 130°C to 150°C.

**A-2.3.2 Calibration Factor or Response Factor**

- Calculate the areas by multiplying peak height by width at half height method, if the unit is not provided with integrator system.
- Calculate the area/mass ( $A/M$ ) ratio by dividing the area of each peak by its mass:

<i>Component</i>	<i>Mass percent</i>	<i>Area</i>	<i>A/M</i>
Xylene	1.0	$A_1$	$A_1/1.0 = K$
Pyridine	1.0	$A_2$	$A_2/1.0 = L$
Alpha picoline	97.0	$A_3$	$A_3/97.0 = M$
Gamma picoline	1.0	$A_4$	$A_4/1.0 = N$

- Set arbitrarily xylene response factor to 1.0 and find response factor of other components as follows:

<i>Components</i>	<i>Slope</i>	<i>Response factor</i>
Xylene	$K/K$	1.0000
Pyridine	$K/L$	Value obtained
Alpha picoline	$K/M$	do
Gamma picoline	$K/N$	do

**A-2.3.3 Elution Order** — Elution order of the component is xylene, pyridine, alpha picoline and gamma picoline.

**A-2.3.4 Test Sample** — Inject 0.5 microlitre of the test sample into gas chromatograph and get the chromatograms within the chart range by means of proper attenuation system.

Calculate the areas of the peaks and multiply the calculated areas by their relative response factors to get the true areas of the peaks. Add up the areas to get the total true area.

#### A-2.4 Calculation

$$\begin{array}{l} \text{Component 'n' in the sample,} \\ \text{percent by mass} \end{array} = \frac{A_n \times 100 - m}{A_t}$$

where

$A_n$  = area of component 'n',

$m$  = percentage of water in the sample, and

$A_t$  = total true area.

### A-3. DETERMINATION OF BOILING RANGE

**A-3.1 Procedure** — Determine the boiling range by the procedure as prescribed in IS : 5298-1969\* applying the following corrections.

#### A-3.1.1 Correction of Thermometer Reading

**A-3.1.1.1 Error of scale** — In all thermometer readings, make the corrections as indicated on the certificate of the instrument.

**A-3.1.1.2 Correction for barometric pressure** — If the barometric pressure prevailing during the determination is 760 mmHg, no correction need be applied to the specified temperature and the thermometer scale as corrected for error of scale may be used as such. If, however, the prevailing barometric pressure deviates from 760 mmHg, the specified temperature shall also be corrected as follows:

- a) For every 27 mmHg above 760 mmHg, subtract 1°C from the specified temperature; and
- b) For every 27 mmHg below 760 mmHg, add 1°C to the specified temperature.

NOTE — These corrections are valid only for pressures above 700 mmHg.

### A-4. DETERMINATION OF MOISTURE

**A-4.0 General** — Moisture is determined by the Karl Fischer method.

**A-4.1 Procedure** — Take 20 g of the material, weigh accurately and determine the moisture content by the procedure given in IS : 2362-1973†.

\*Determination of distillation range and of distillation yield.

†Determination of water by Karl Fischer method ( *first revision* ).

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**A-5. TEST FOR SOLUBILITY**

**A-5.1** Mix 10 parts of the material in 90 parts of water and shake the mixture. The material shall be clear and homogeneous.

**A P P E N D I X   B**

*( Clause 5.1 )*

**SAMPLING OF ALPHA PICOLINE**

**B-1. GENERAL REQUIREMENTS FOR SAMPLING**

**B-1.1** Samples shall be taken in a protected place not exposed to damp air, dust or soot.

**B-1.2** The sampling instrument shall be clean and dry.

**B-1.3** Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

**B-1.4** To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

**B-1.5** The samples shall be placed in suitable, clean, dry and airtight glass bottles or other suitable containers on which the material has no action.

**B-1.6** The sample containers shall be of such a size that they are almost three-fourth filled by the sample.

**B-1.7** Each sample container shall be sealed airtight after filling, and marked with full details of sampling, the date of sampling and details given under 4.2.

**B-2. SCALE OF SAMPLING**

**B-2.1** Lot — All the containers in a single consignment of the material of the same grade and drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

**B-2.2** For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately.

**B-2.3** The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

**TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED  
FROM LOTS OF DIFFERENT SIZES**

( Clause B-2.3 )

LOT SIZE	SAMPLE SIZE
(N)	(n)
(1)	(2)
3 to 15	3
16 „ 40	4
41 „ 110	5
111 „ 180	6
181 „ 300	7
301 „ 500	8
501 and above	9

**B-2.4** The containers shall be chosen at random from the lot with the help of a suitable random number table. Reference may be made to IS : 4905-1968\* for guidance to random selection procedures.

### **B-3. TEST SAMPLE AND REFEREE SAMPLE**

**B-3.1** From each of the containers selected as in **B-2.3**, draw with the help of a sampling bottle a representative portion of the material from different part of the container. Out of this portion from each container equal quantity of the material shall be taken and thoroughly mixed to form a composite sample of about 1 500 ml. This composite sample shall be thoroughly mixed and divided into three equal portions, one for the purchaser, another for the supplier and the third for the referee.

**B-3.2** The remaining portion corresponding to each of the selected containers ( **B-2.3** ) shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the  $n$  containers selected shall be for the purchaser, another for the supplier and the third for the referee

**B-3.3** All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in **B-1.7**.

\*Methods for random sampling.

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**B-3.4** The referee samples consisting of a composite sample and a set of  $n$  individual samples, shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. These shall be used in case of any dispute between the two.

**B-4. TESTS**

**B-4.1** Tests for picoline content and moisture shall be conducted on individual samples.

**B-4.2** Tests for the remaining characteristics shall be conducted on the composite sample.

**B-5. CRITERIA FOR CONFORMITY**

**B-5.1 For Individual Samples** — The lot shall be declared as conforming to the requirements of picoline content and moisture if each of the test results on the individual samples satisfies the corresponding requirement of the test.

**B-5.2 For Composite Sample** — For declaring the conformity of a lot to the requirements of all other characteristics tested on the composite sample, the test results shall satisfy the relevant requirements given in 3 and Table 1.

## INDIAN STANDARDS

### ON

### ORGANIC CHEMICALS ( MISCELLANEOUS ) MATERIALS

#### IS:

- 245-1970 Trichloroethylene, technical ( *second revision* )
- 501-1963 Oxalic acid, technical and analytical reagent ( *revised* )
- 716-1970 Pentachlorophenol ( *first revision* )
- 717-1969 Carbon disulphide, technical ( *first revision* )
- 718-1970 Carbon tetrachloride ( *first revision* )
- 869-1969 Ethylene dichloride ( *first revision* )
- 880-1956 Tartaric acid
- 3321-1973 Formaldehyde solution ( *first revision* )
- 4105-1967 Styrene ( vinyl benzene )
- 4306-1973 Hexamethylenetetramine ( hexamine ) ( *second revision* )
- 4566-1968 Methylene chloride ( dichloromethane ), technical
- 5149-1969 Maleic anhydride, technical
- 5158-1969 Phthalic anhydride, technical
- 5254-1969 Acetanilide
- 5271-1969 Paraformaldehyde
- 5295-1969 Ethylene glycol
- 5296-1969 Chloroform, technical and analytical
- 5297-1969 Perchloroethylene ( tetrachloroethylene ), technical
- 5341-1969 Benzyl chloride, technical
- 5464-1970 Citric acid, monohydrate
- 5573-1969 Ethylene oxide
- 5591-1969 Chlorobenzene
- 5592-1969 Monochloroacetic acid
- 5992-1969 *p*-Dichlorobenzene, technical
- 6393-1971  $\alpha$ -Phenylacetamide
- 6412-1971 Benzyl chloride, technical
- 6515-1972 Sodium pentachlorophenate, technical
- 6712-1972 *o*-Dichlorobenzene
- 6716-1972 Benzoic acid, technical
- 6718-1972 Phenoxyacetic acid
- 6768-1973 *m*-Aminophenol
- 6775-1973 Ethyl chloride, technical
- 6971-1973 2-ethyl hexan-1-ol
- 6972-1973 Benzotrichloride, technical
- 7134-1973 Diphenyl
- 7135-1973 Dimethyl sulphate, technical
- 7220-1974 Ethylenediaminetetra-acetic acid, pure and technical



7330-1974 **Methods of test for ion-exchange resins**  
7559-1974 Salicylic acid  
7618-1974 Hexachloroethane  
7619-1974 Pentaerythritol  
7729-1975 Sodium monochloroacetate  
7901-1975 Triethanolamine, technical  
7910-1975 Monoethanolamine  
7911-1975 Diethanolamine  
7918-1975 Diethylene glycol

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Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

**Telephone : 27 01 31 (20 lines)**

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#### Regional Offices

		Telephone
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